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THE CORROSION PROTECTION OF METALS BY
ION VAPOR DEPOSITED ALUMINUM

By M.D. Danford

Materials and Processes Laboratory
Science and Engineering Directorate

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George C. Marshall Space Flight Center

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TECHNICAL MEMORANDUM

THE CORROSION PROTECTION OF METALS BY ION VAPOR DEPOSITED ALUMINUM

INTRODUCTION

Because of increasing interest in the ion vapor deposition (IVD) method, it seemed worthwhile to investigate the corrosion protection afforded various substrate metals by IVD aluminum (Al). Here the aluminum coat acts as a sacrificial anode, which should provide corrosion protection to the underlying substrate metal. The Al coat in this case was type 1100 Al, which is 99.99-percent pure Al metal.

In addition to corrosion protection, hard anodized IVD Al provides an extremely hard, wear-resistant coat. It is used in steam turbines and on the roots of turbine blades for corrosion protection. It is also used on fasteners to prevent galvanic interaction. Other uses include aircraft landing gear, the springs of the auxiliary power unit (APU) of the space shuttle solid rocket boosters, and military use of rocket launchers and tanks.

EXPERIMENTAL

Flat plates, 10.2 by 15.2 cm (4 by 6 in), were coated with IVD Al. Three 2219-T87 Al plates were studied. The first of these had a 51-micron (2-mil) IVD anodized coat. A second had a 25.4-micron (1-mil) IVD Al coat, and a third had a 25.4-micron (1-mil) IVD anodized coat. Three plates with a base metal of Ti-6Al-4V were also studied in a similar manner. In addition, a 4130 steel plate with an IVD Al coat of 25.4 microns (1 mil) was studied. Similar studies were carried out for a D6AC steel plate and a 4340 steel plate. Two 7075-T6 Al plates—one with a 25.4-micron (1-mil) IVD Al coat, and the other with a 51-micron (2-mil) anodized IVD Al coat—were also studied. In the case of IVD Al coats that were not anodized, dichromate conversion coats were used on top to increase corrosion resistance.

Since anodized IVD Al coats were too hard to remove by sanding, this coating was removed from one side of each plate by grit blasting to provide electrical contact. The anodized sides were wiped with alcohol to remove fingerprints. They were then clamped into flat corrosion cells manufactured by EG&G-PARC and exposed to 3.5-percent sodium chloride (NaCl) at pH 5.4, a very corrosive medium. Corrosion data were obtained over a period of 27 days. Silver/silver chloride reference electrodes were used in all cases.

Both electrochemical impedance spectroscopy (EIS), an alternating current (ac) method, and the polarization resistance technique (PR), a direct current (dc) method, were employed in this investigation. The EIS method was employed primarily for those cases in which the IVD Al coat was hard anodized, while the PR method was used where there was no anodizing, but only a dichromate conversion coat applied to the IVD Al.

The EG&G-PARC model 388 ac impedance system was used for all corrosion measurements. For the EIS measurements, data were taken in three sections. The first two sections, beginning at

0.001 Hz and 0.1 Hz, respectively, were obtained using the fast Fourier transform technique. The third data section, ranging from 6.28 to 40,000 Hz, was collected using the lock-in amplifier technique. The sequencing was performed automatically using the autoexecute procedure, with all data merged to a single set for each run. After collection, these data were processed and analyzed by computer using the models of figures 1 and 2. The same computer also controlled the experiment.

Data for the PR technique were collected using the same instrumentation with the EG&G-PARC model 352 software, which was developed especially for dc measurements. Instrumentation developed by EG&G-PARC automatically corrected the data for IR drop during the scan. The potential applied to the specimen during the scan was varied from -20 mV to +20 mV on either side of the corrosion potential E_{CORR} , with data points (current and potential) being recorded in 1/4-mV increments.

The primary equivalent circuit model for interpretation of EIS data is shown in figure 1. The circuit model of figure 2 was used to calculate the effect of diffusion polarization. The Warburg coefficient sigma is obtained using this model. The higher the value of sigma, the less the diffusion of the surrounding medium through the specimen coat. If the value of sigma exceeds that of the charge transfer parameter $R(T)$, the corrosion mechanism is diffusion controlled. The development and selection of the models of figures 1 and 2 have been discussed previously.²

Values for each of the circuit components in either figure 1 or 2 were treated as parameters in the nonlinear ORGLS³ least-squares program, which automatically adjusted these parameters to obtain a best fit to the observed Bode magnitude data (log impedance versus log ω ($\omega = 2\pi \times$ frequency)). Good estimates of the corrosion rates were obtained from EIS data using the Stern-Geary equation for charge transfer control.⁴⁻⁶ Tafel constants (b_a and b_c) were assumed to be 50 mV each. The value of 50 mV for each of the Tafel constants has been found to provide excellent agreement with values of I_{CORR} obtained by the dc PR measurements.⁷ Corrosion rates were determined from the I_{CORR} values by methods described previously.⁷

In the PR method, curves of potential versus current were obtained, the data were analyzed using the program POLCURR.⁸ The theory for the PR techniques has been described previously.⁹ This method has an advantage in that values for the Tafel constants are obtained directly from the experimental data and are not assumed as in the case of EIS experiments. However, more mechanistic information is obtainable in EIS experiments.

RESULTS AND DISCUSSION

Ti-6Al-4V Base Metal

51-Micron (2-mil) Anodized IVD Al Coat. Only the EIS method was used in this case. Corrosion currents were generally too small to be measured by the PR method. The charge transfer resistance, $R(T)$ -time curve (fig. 3) shows an increasing trend in the value of $R(T)$, except for the last 4 days. The corrosion rate is inversely proportional to the value of $R(T)$. The pore resistance, $R(P)$ -time curve, is shown in figure 4. It generally follows the trends of the $R(T)$ -time curve and shows that the resistance of the anodized coating to diffusion of the medium is quite good. The corrosion current, I_{CORR} -time curve, as obtained from the EIS data, is shown in figure 5. As expected from the $R(T)$ -time curve, I_{CORR} decreases with time, except for the last few days. The Warburg coefficient, sigma-time curve (fig. 6), is consistent with trends observed in the $R(T)$ -time and $R(P)$ -time curves.

The 7-day average corrosion rate was 0.000608 mils per year (mil/yr); and the 27-day average corrosion rate was 0.0003114 mil/yr, a decrease of 50.3 percent. The corrosion rate of bare Ti-6Al-4V is 0.00037 mil/yr and is thus as small as any of the corrosion rates observed for the anodized IVD Al coated samples. The mean daily change in the value of I_{CORR} , as determined by linear regression, was 0.0 μ A/day, indicating little overall change in this time period. The average corrosion currents for the 7- and 27-day time periods are shown in table 1 for all samples, together with the mean daily change in I_{CORR} . Metallographic examination verified the 51-micron (2-mil) thick IVD Al coat, with the thickness of the anodized layer being 18 microns (0.7 mil). A photograph of the spot exposed to the corrosive medium for 27 days shows excellent resistance to corrosion (fig. 7).

25-Micron (1-mil) Anodized IVD Al Coat. Because this specimen showed a large drop in the value of $R(T)$ (183.6 k-ohms), as compared to that of the 2-mil anodized IVD coat (2,591.0 k-ohms), it was necessary to do an EIS run only on the first day. Values of $R(P)$ and the Warburg coefficient showed similar drops. The corrosion current was measurable with the PR technique; therefore, this method was used to make subsequent measurements of I_{CORR} . The I_{CORR} -time curve is shown in figure 8 and shows a rather level oscillating behavior. The 7-day average corrosion rate was 0.0064 mil/yr and the 27-day average was 0.0066 mil/yr, which represents an increase of only 2.8 percent. Linear regression analysis gave a mean daily change of only -0.0001 μ A/day. Metallographic examination of the cross section verified the IVD Al thickness of 25 microns (1 mil), but showed that the thickness of the anodized layer was only 10 microns (0.4 mil). This probably accounts for most of the difference in corrosion rates between the 25-micron (1-mil) anodized IVD Al and the 51-micron (2-mil) anodized IVD Al coat. A photograph (not shown) of the corrosion spot showed only a slight degradation of the anodized coat after the 27-day exposure.

25-Micron (1-mil) IVD Al Coat. Corrosion testing of this sample was limited to the PR method, since the corrosion current was quite high and amenable to these measurements. The I_{CORR} -time curve for this specimen is shown in figure 9. It is generally level, though oscillating in nature. The 7-day average corrosion rate was 0.3307 mil/yr, and that for the 27-day period was 0.3492 mil/yr—an increase of 5.6 percent. These values are much higher than those for the anodized samples. The mean daily increase in the corrosion rate was 0.0032 μ A/day, also higher than those for the anodized samples. Examination of the corrosion spot showed that the bare Ti-6Al-4V base metal was exposed after the 27-day period. The corrosion rate for this case is thus quite significantly higher than that for the base metal (0.00037 mil/yr). However, corrosion protection of the base metal does occur due to the cathodic protection provided by the sacrificial IVD Al coat.

4130 Steel Base Metal

This metal was coated with a 25-micron (1-mil) thickness of IVD Al and was covered by a dichromate conversion coat. Since corrosion currents were large, only the PR method was used for study of this sample. The I_{CORR} -time curve is shown in figure 10. The 7-day average corrosion rate was 0.8104 mil/yr, and the 27-day average was 1.46 mil/yr—an increase of 80 percent. These corrosion rates are quite low, however, when compared to that of the bare metal in the same medium (18.3 mil/yr). Thus, the IVD Al coat is quite effective in the corrosion protection of this metal. The mean daily increase in I_{CORR} was 0.0981 μ A/day, significantly larger than those observed for the anodized specimens previously discussed. Examination of the corrosion spot showed that considerable corrosion of the IVD Al coat had occurred, with the bare 4130 metal base being exposed in one or two spots.

D6AC Steel Base Metal

Coating coverage for this metal was the same as that for 4130 steel. Again, only the PR method was used for corrosion studies. The I_{CORR} -time curve for this material is shown in figure 11, and exhibits a gradually increasing trend. The 7-day average corrosion rate was 0.0678 mil/yr, and that for the 27-day period was 0.212 mil/yr—an increase of 212.5 percent. The corrosion rate for the base metal in the same medium was 66.3 mil/yr, and shows again that the IVD Al coat provides excellent corrosion protection. The mean daily increase in I_{CORR} was 0.032 $\mu\text{A/day}$. Examination of the corrosion spot again showed extensive corrosion of the IVD Al coat; nevertheless, corrosion protection was quite effective.

4340 Steel Base Metal

The coating coverage for this steel was the same as for the 4130 and D6AC steels. The I_{CORR} -time curve is shown in figure 12 and is highly oscillatory in nature. The 7-day average corrosion rate was 0.2903 mil/yr, and the 27-day average was 0.5328—an 83.4-percent increase. The corrosion rate for the base metal in the same medium was 31.6 mil/yr, much higher than the values observed for the protected metal. The mean daily increase in I_{CORR} was 0.0483 $\mu\text{A/day}$. Examination of the corrosion spot showed that the IVD Al coat was badly corroded, with corrosion depth penetrating the base metal. However, corrosion protection of the base metal is provided by the IVD Al coat which maintains its cathodic nature.

2219-T87 Al Base Metal

51-Micron (2-mil) Anodized IVD Al Coat. Both the EIS and PR methods were used for this case. Values of $R(T)$ and $R(P)$ were much lower than for Ti-6Al-4V, and values for the corrosion current were appreciably higher. The $R(T)$ -time curve (fig. 13) exhibits a generally decreasing value of $R(T)$. Similar behavior exists for the $R(P)$ -time curve in figure 14. The I_{CORR} -time curve (fig. 15) generally increases, while the Warburg coefficient-time curve (fig. 16) remains at about the same level throughout the test. The 7-day average corrosion rate was 0.0230 mil/yr and the 27-day average was 0.0456 mil/yr, an increase of 98.3 percent. The mean daily increase in I_{CORR} was 0.0109 $\mu\text{A/day}$, considerably higher than for the Ti-6Al-4V case. The average corrosion rates are quite low, much lower than the 0.771 mil/yr measured for the bare metal, showing that considerable corrosion protection still exists. A photograph of the exposed spot in figure 17 shows that considerable corrosion of the anodized protective layer occurred, with depth of corrosion extending to the bare metal surface. A cross section of the anodized plate verified the 51-micron (2-mil) thickness of the IVD Al coat, with the thickness of the anodized layer being 20 microns (0.8 mil).

25-Micron (1-mil) Anodized IVD Al Coat. Since the corrosion current was high enough to be measured by the PR method, this technique was used for the measurement of I_{CORR} throughout. However, an EIS measurement taken on the first day of exposure revealed values of $R(T)$ and $R(P)$ to be considerably higher than those for the 51-micron (2-mil) anodized case. The I_{CORR} -time curve is shown in figure 18. This oscillating curve is generally flat, with a large peak occurring at 20 days. The 7-day average corrosion rate was 0.00347 mil/yr, while that for the 27-day period was 0.00561 mil/yr—an increase of 61.7 percent. Linear regression analysis revealed a mean daily increase in I_{CORR} of 0.0003 $\mu\text{A/day}$. These parameters are actually much better than those of the 51-micron (2-mil) case. A cross section of this sample verified the 25-micron (1-mil) IVD coating thickness, but revealed a thickness of

only 15 microns (0.6 mil) for the anodized layer. This thickness is actually less than that for the 51-micron (2-mil) case. Therefore, based on these results, the quality of the 25-micron anodized coat appears to be superior to the 51-micron anodized coat. The appearance of the corrosion spot was also much improved over that for the 51-micron (2-mil) case.

25-Micron (1-mil) IVD Al Coat. Corrosion currents were very high, and this sample was studied by only the PR method. The I_{CORR} -time curve is shown in figure 19, and generally increases with time. The 7-day average corrosion rate was 0.244 mil/yr, while that for the 27-day average was 0.553 mil/yr—an increase of 126.4 percent. The mean daily increase in I_{CORR} was 0.634 $\mu\text{A/day}$, as determined by linear regression. These values are thus much greater than those for the anodized specimens. Corrosion of the IVD Al layer was quite extensive. The 27-day average corrosion rate is approaching that for bare 2219-T87 Al (0.771 mil/yr). Corrosion protection provided by the anodized samples is thus much superior to that provided by this specimen.

7075-T6 Al Base Metal

25-Micron (1-mil) IVD Al Coat. 7075-T6 Al is much more susceptible to corrosion than 2219-T8 Al. The corrosion rate for the base metal is 10.1 mil/yr in the medium employed in this work compared to a corrosion rate of only 0.771 mil/yr for 2219-T87 Al. For this reason, both anodized and unanodized coated specimens were studied. Since corrosion currents were amenable to study by the PR method, this technique was employed throughout. The I_{CORR} -time curve is shown in figure 20 and is highly oscillatory in nature, with three peaks evident. The 7-day average corrosion rate was 0.3678 mil/yr, and the 27-day average was 1.466 mil/yr—an increase of 298.6 percent. These rates, although high, are significantly less than that for the base metal. The mean daily increase in I_{CORR} was 0.1298 $\mu\text{A/day}$, the highest value observed for any of the metals in this work. Examination of the corrosion spot revealed that corrosion penetration to the base metal had occurred in one spot. Thus, although corrosion currents are high, a high degree of corrosion protection was evident in this case.

51-Micron (2-mil) Anodized IVD Al Coat. A cross section of this sample showed the thickness of the IVD Al coat to be 66 microns (2.6 mils), exceeding that of the expected 51-micron (2.0-mil) thickness. The thickness of the anodized layer was 38 microns (1.5 mils), much thicker than those for the other anodized coats. Although corrosion currents became quite small after the first day, they were still measurable by the PR method, so that this was the only technique employed. An EIS measurement on the first day produced rather high values for $R(T)$ and $R(P)$, indicating that corrosion protection was quite good.

The I_{CORR} -time curve is shown in figure 21. Outside of day 1, it exhibits very low values. The 7-day average corrosion rate was 0.00882 mil/yr, and the 27-day average was 0.0030627 mil/yr—a decrease of -65.3 percent. The mean daily change in I_{CORR} was -0.001 $\mu\text{A/day}$. Thus, the corrosion rate actually decreases with time and verifies the effectiveness of the anodized layer in providing corrosion protection. Examination of the corrosion spot showed little damage by corrosion.

CONCLUSIONS

Anodized IVD Al coatings, in addition to providing a very hard, wear resistant surface, provided the best corrosion protection to substrate metals studied (2219-T87 Al; 7075-T6 Al; Ti-6Al-4V; and

4130, 4340, and D6AC steels). Cross sectioning of the anodized IVD Al coats verified that the thickness of the IVD deposited Al coats was under excellent control. However, the thickness and quality of the anodized layers varied, sometimes considerably, from sample to sample.

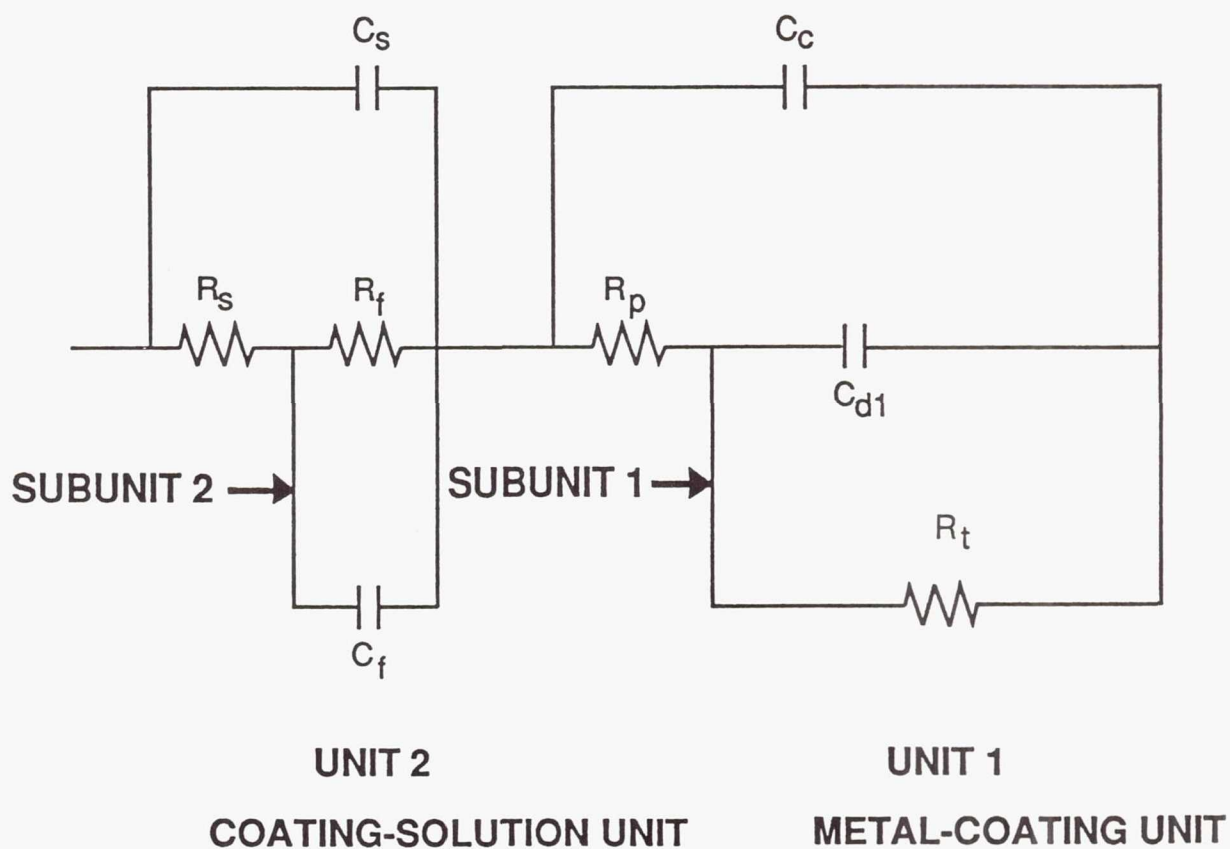
Metals coated with IVD Al which was not anodized, but covered by a dichromate conversion coat, also showed enhanced corrosion protection for the substrate metal. In the very corrosive medium employed in this work, corrosion of the IVD Al coats was extensive, but corrosion rates were far below those observed for the substrate metals of the 4130, 4340, and D6AC steels studied here. The IVD Al coat acted as a sacrificial anode and provided cathodic protection to the substrate metal. In the case of Ti-6Al-4V, the corrosion rate of IVD Al coating was much higher than the substrate, but the IVD Al coating did provide cathodic protection to the substrate. The general conclusion is that IVD Al coats, both anodized and unanodized, provide good corrosion protection to substrate metals.

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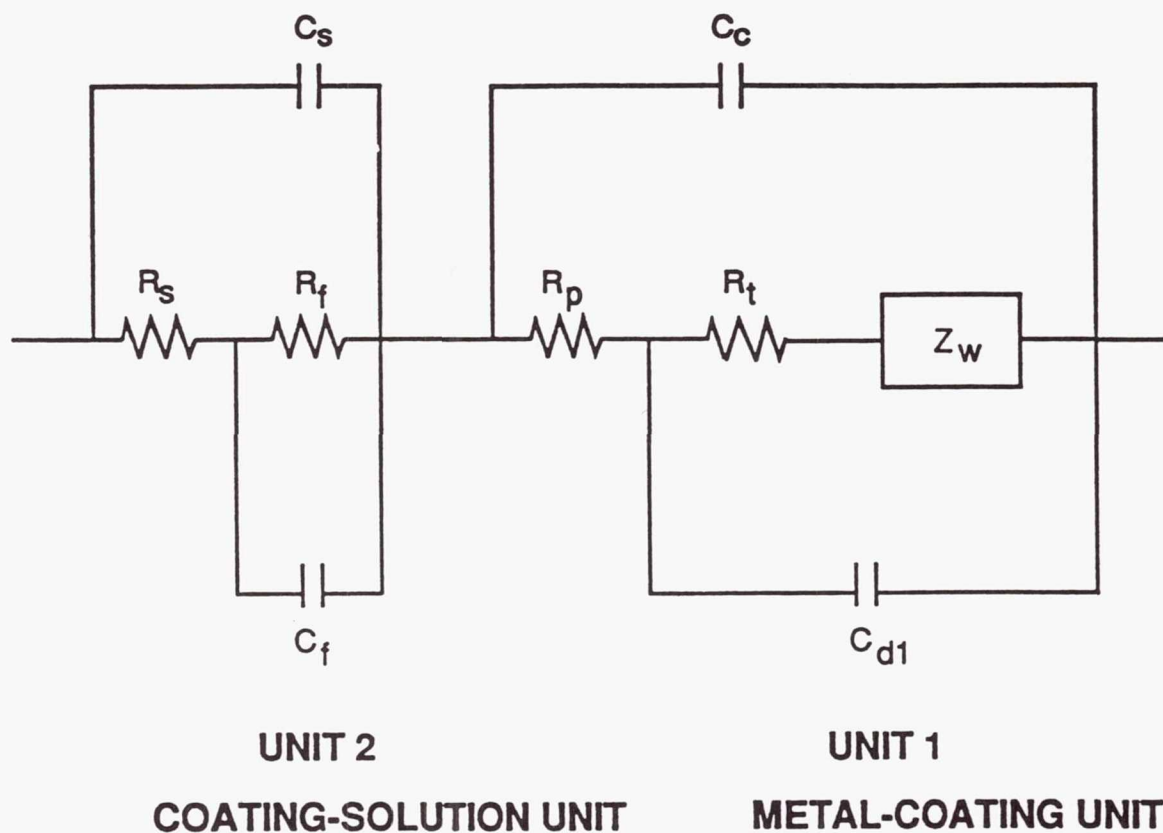
Table 1. Average corrosion currents for IVD coated metals with mean daily increase.

Material and Coat	7-Day Average (μA)	27-Day Average (μA)	Percent Increase	Mean Daily Increase (μA)
1. 2219-T87 Al, 2 mil Anodized IVD	0.0522	0.1037	98.3	0.0109
2. Ti-6Al-4V, 2 mil Anodized IVD	0.0027	0.0006	-50.3	0.0
3. 2219-T87 Al, 1 mil Anodized IVD	0.0079	0.0127	61.7	0.0003
4. Ti-6Al-4V, 1 mil Anodized IVD	0.0145	0.014	2.8	-0.0001
5. Ti-6Al-4V, 1 mil IVD	0.7514	0.7933	5.6	0.0032
6. 4130 Steel, 1 mil IVD	1.8395	3.3111	80.0	0.0981
7. D6AC Steel, 1 mil IVD	0.1540	0.4812	212.5	0.0320
8. 4340 Steel, 1 mil IVD	0.6590	1.2085	83.4	0.0483
9. 2219-T87 Al, 1 mil IVD	0.5483	1.2411	126.4	0.0634
10. 7075-T6 Al, 1 mil IVD	0.7980	3.1809	298.6	0.1298
11. 7075-T6 Al, 2 mil Anodized IVD	0.01914	0.00665	-65.3	-0.001



- C_s SOLUTION CAPACITANCE
- R_s SOLUTION RESISTANCE
- C_f FARADAIC CAPACITANCE (COATING/SOLUTION)
- R_f FARADAIC RESISTANCE
- C_c COATING CAPACITANCE
- R_p COATING RESISTANCE
- R_t CHARGE TRANSFER RESISTANCE
- C_{d1} METAL/COATING INTERFACE CAPACITANCE

Figure 1. Primary equivalent circuit model for analysis of EIS data.



- C_s SOLUTION CAPACITANCE
- R_s SOLUTION RESISTANCE
- C_f FARADAIC CAPACITANCE (COATING/SOLUTION)
- R_f FARADAIC RESISTANCE
- C_c COATING CAPACITANCE
- R_p COATING RESISTANCE
- R_t CHARGE TRANSFER RESISTANCE
- C_{d1} METAL/COATING INTERFACE CAPACITANCE
- Z_w WARBURG IMPEDANCE (DIFFUSION POLARIZATION)

Figure 2. Equivalent circuit model for calculating the Warburg coefficient.

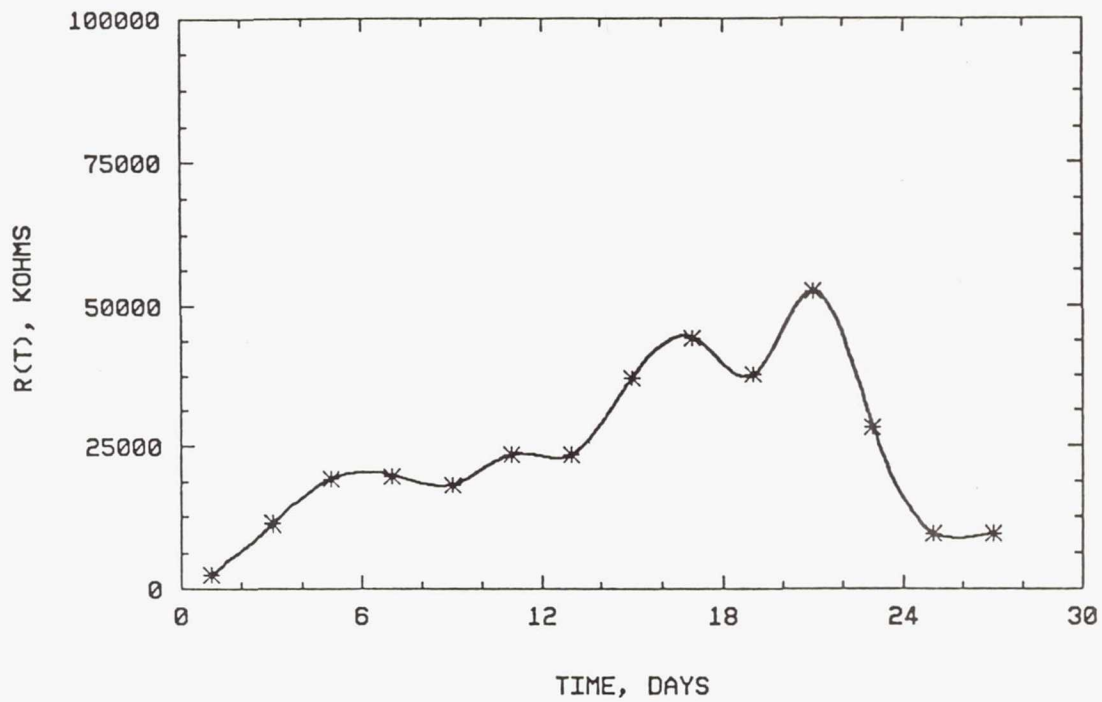


Figure 3. Charge transfer resistance, Ti-6Al-4V, 2-mil anodized coat.

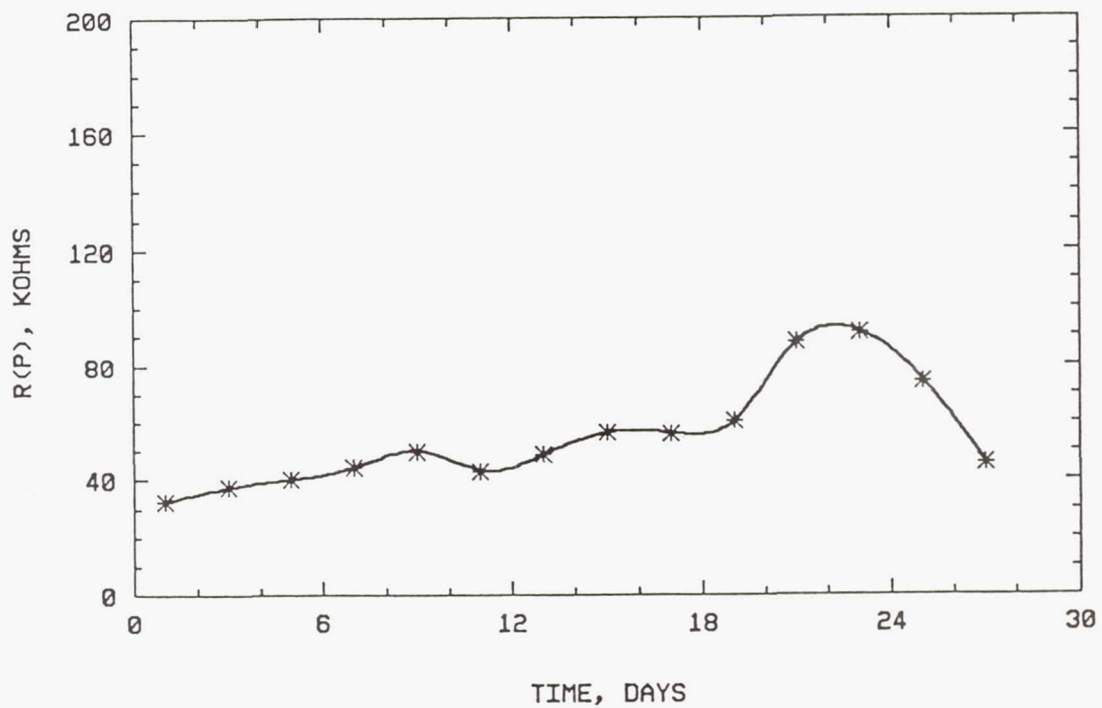


Figure 4. Pore resistance, Ti-6Al-4V, 2-mil anodized coat.

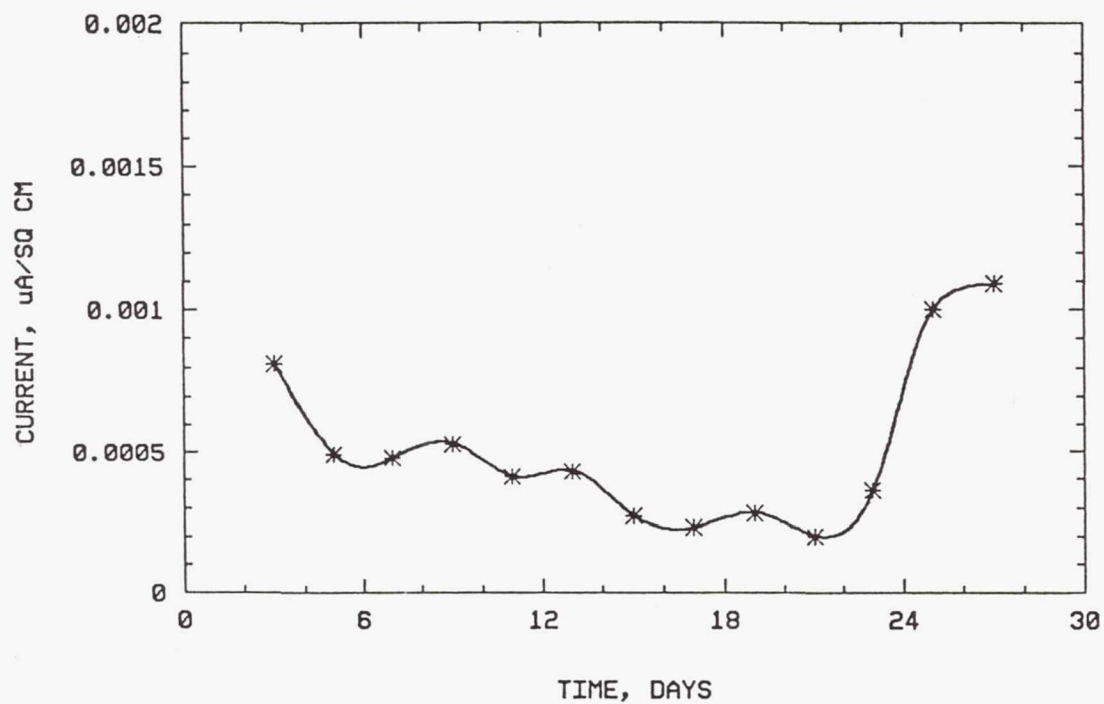


Figure 5. Corrosion current, Ti-6Al-4V, 2-mil anodized coat.

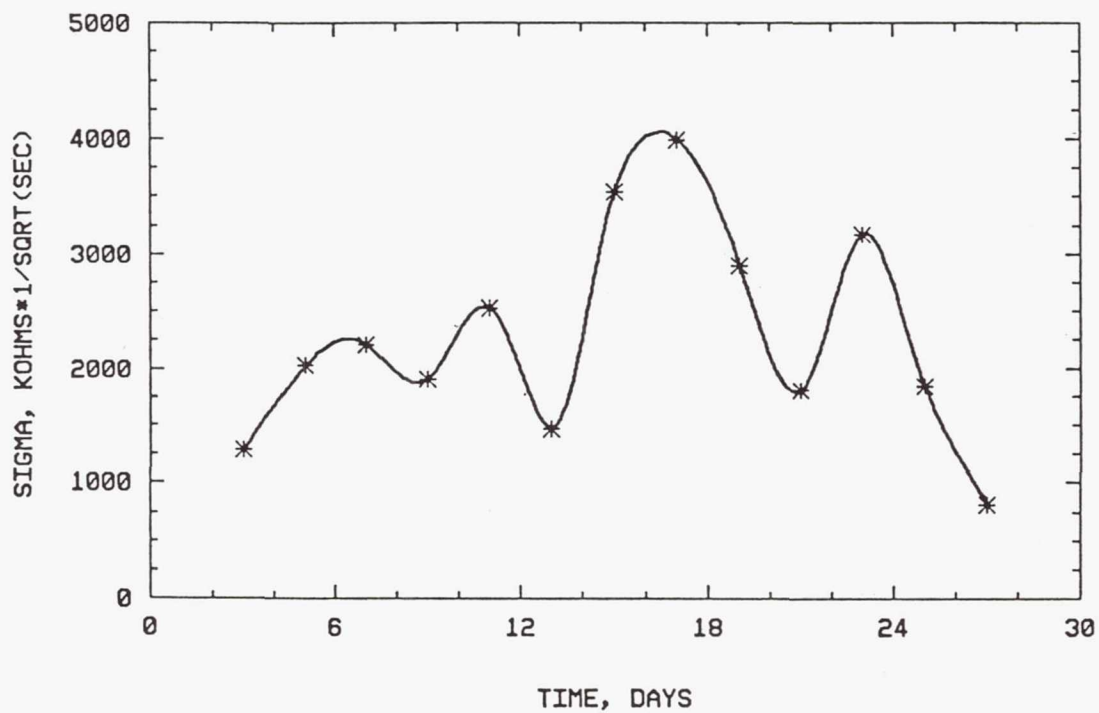


Figure 6. Warburg coefficient, Ti-6Al-4V, 2-mil anodized coat.

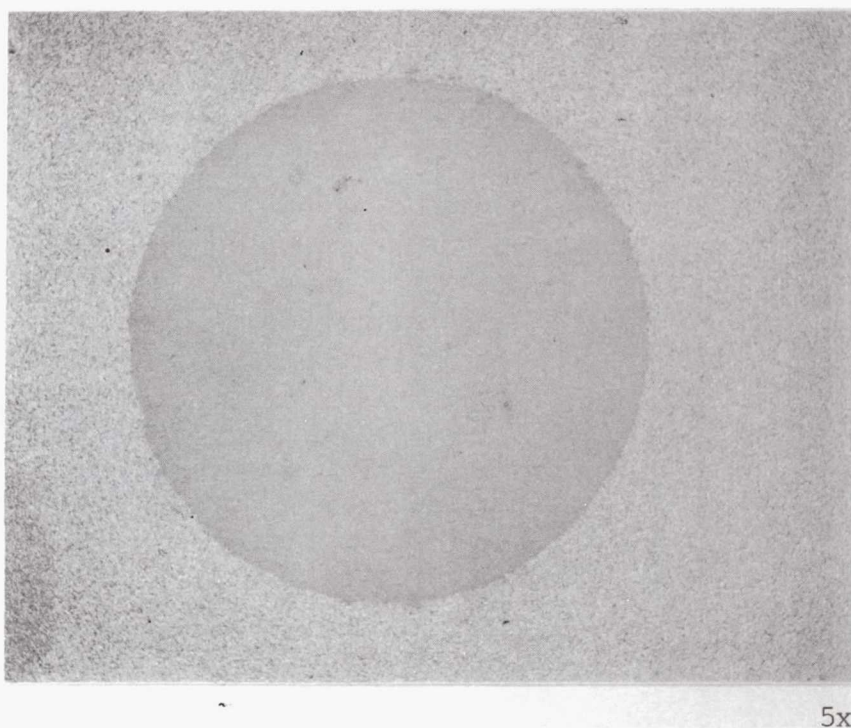


Figure 7. Ti-6Al-4V (2-mil anodized IVD coat). Corrosion after 27-days exposure to 3.5-percent NaCl.

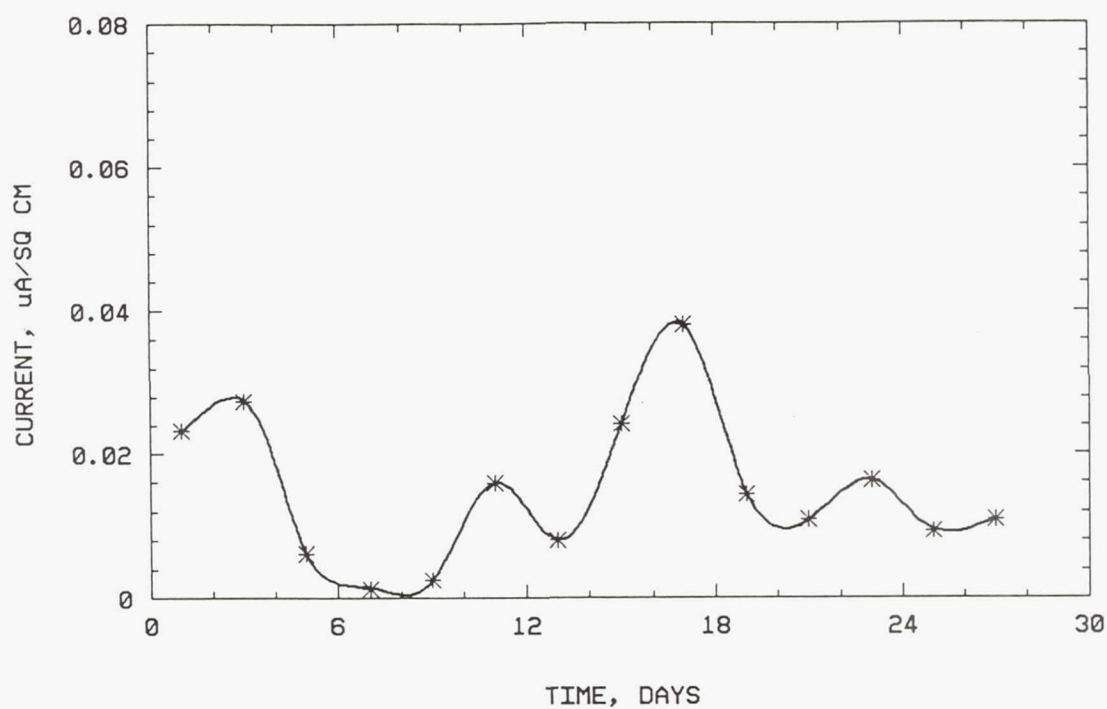


Figure 8. Corrosion current, Ti-6Al-4V, 1-mil anodized IVD coat.

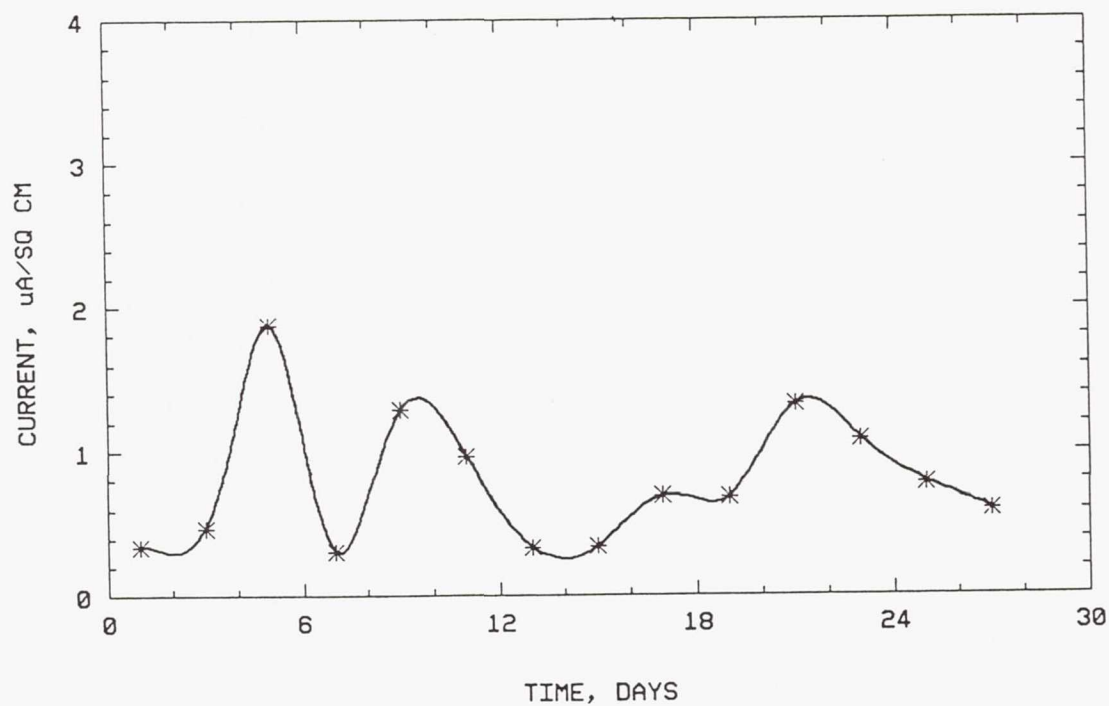


Figure 9. Corrosion current, Ti-6Al-4V, 1-mil IVD coat.

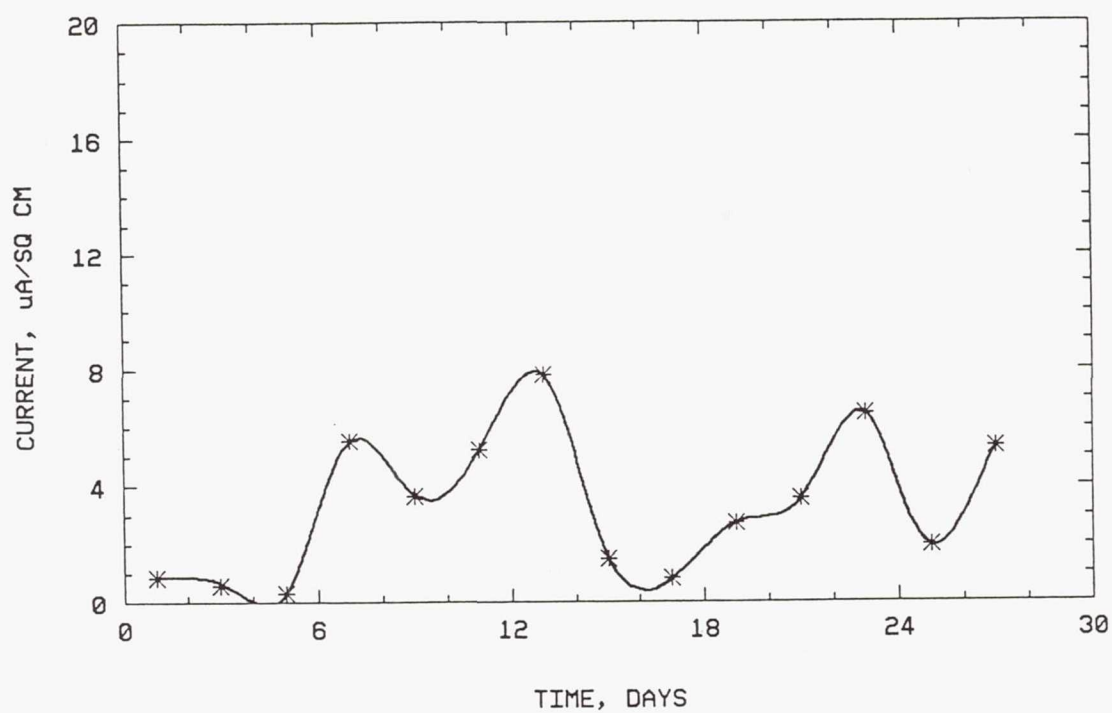


Figure 10. Corrosion current, 4130 steel, 1-mil IVD coat.

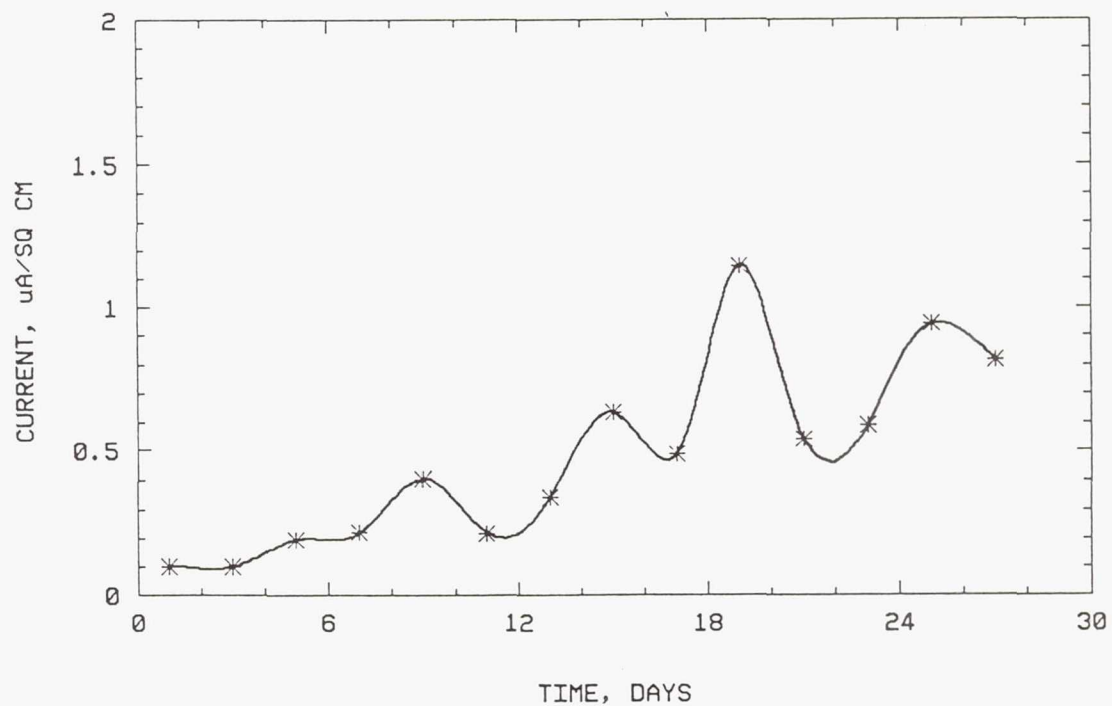


Figure 11. Corrosion current, D6AC steel, 1-mil IVD coat.

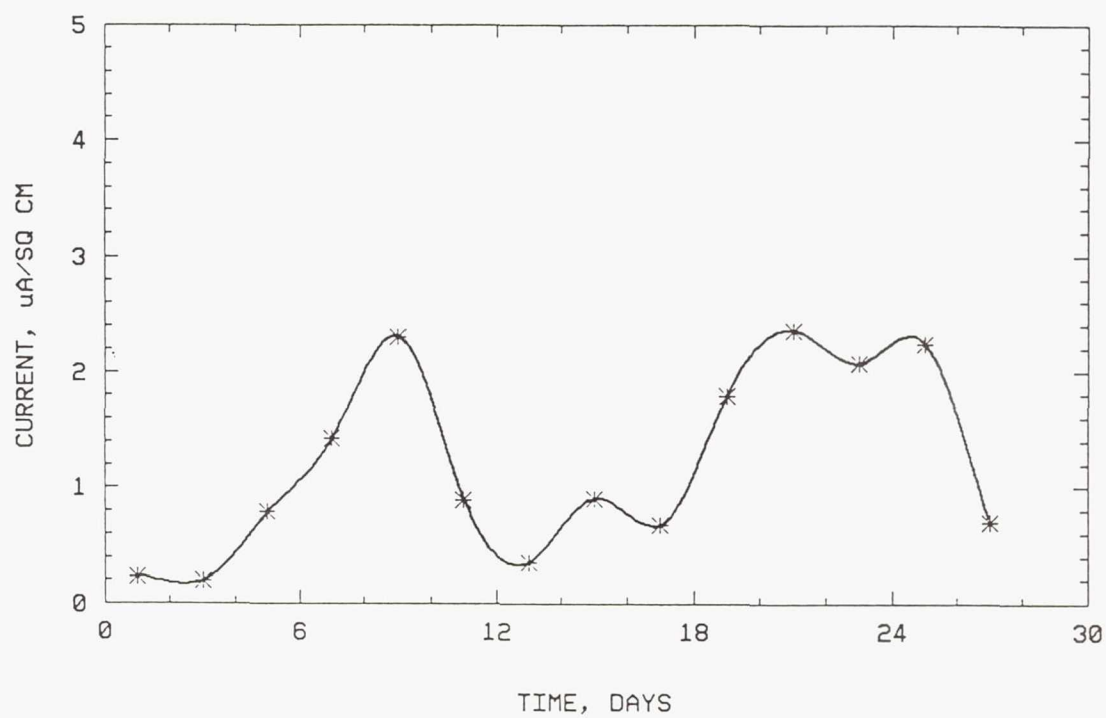


Figure 12. Corrosion current, 4340 steel, 1-mil IVD coat.

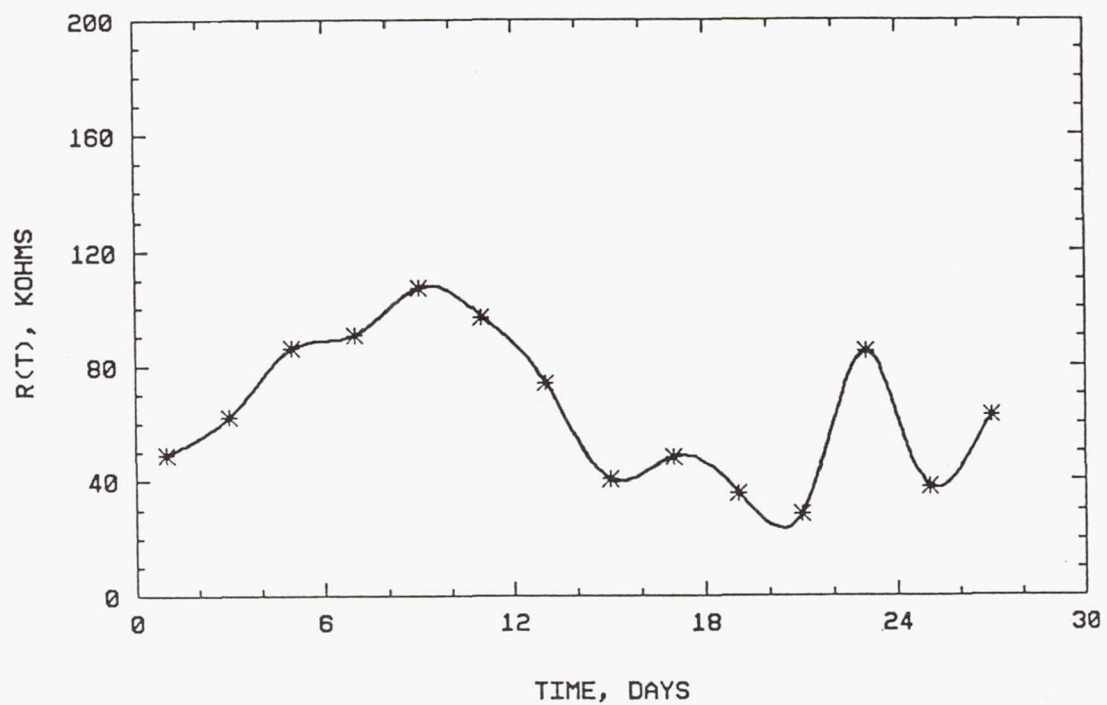


Figure 13. Charge transfer resistance, 2219 Al, 2-mil anodized coat.

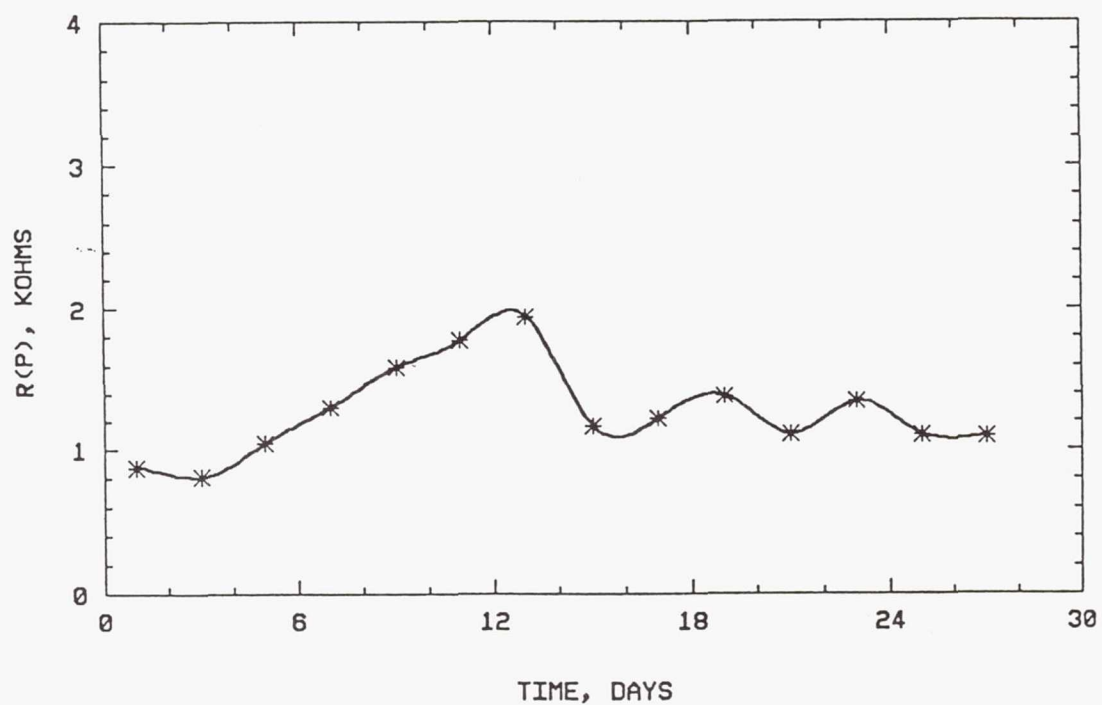


Figure 14. Pore resistance, 2219 Al, 2-mil anodized coat.

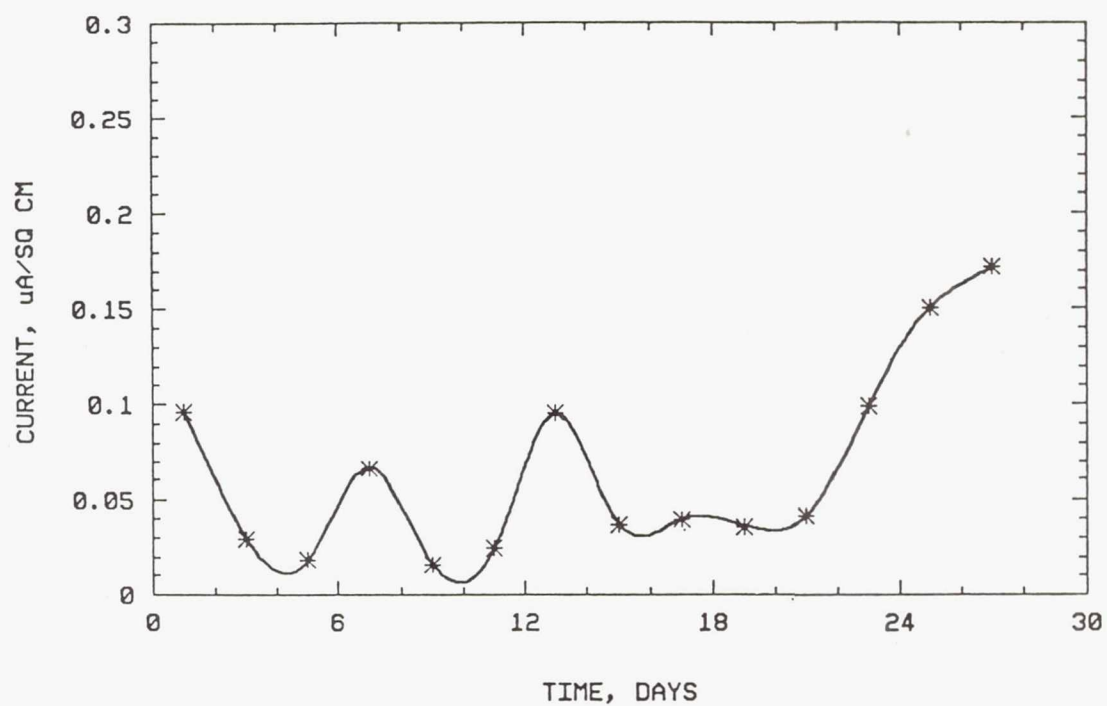


Figure 15. Corrosion current, 2219 Al, 2-mil anodized coat.

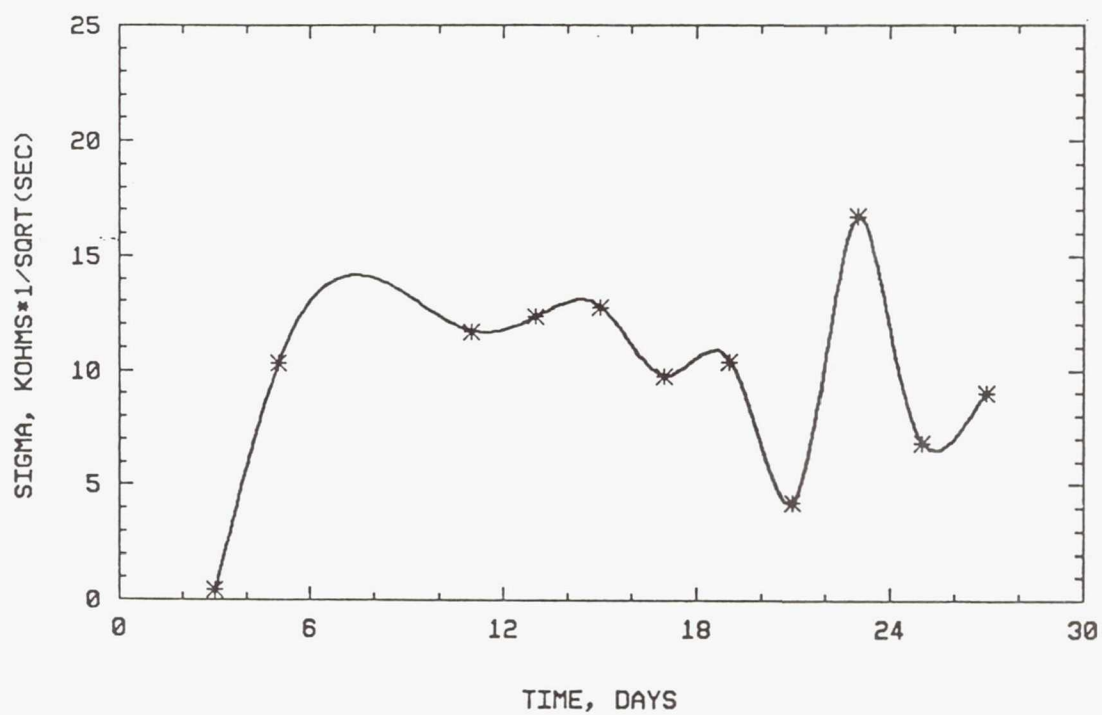


Figure 16. Warburg coefficient, 2219 Al, 2-mil anodized coat.

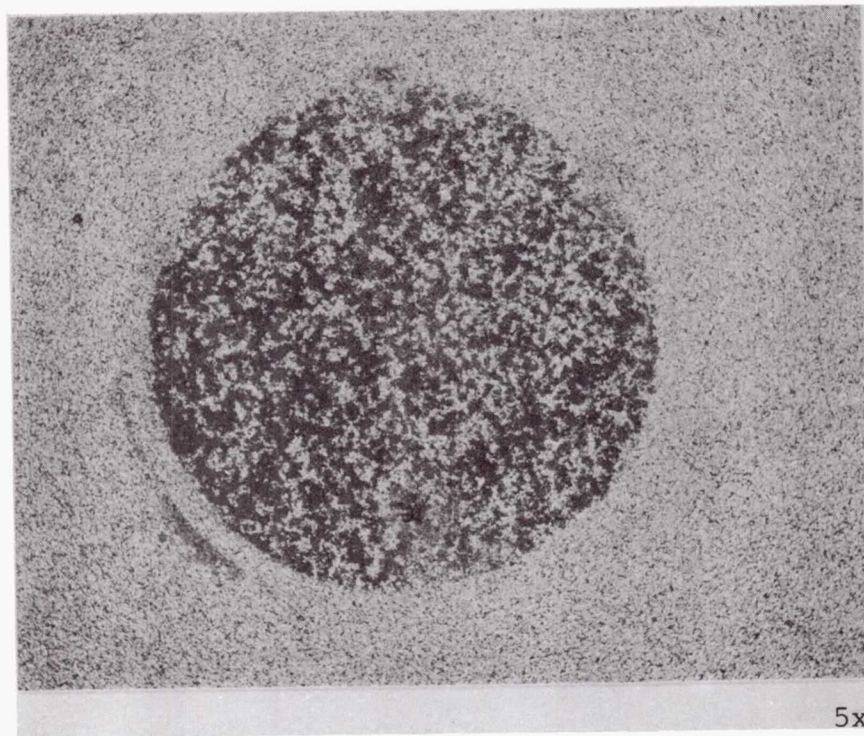


Figure 17. 2219-T87 Al (2-mil anodized IVD coat). Corrosion after 27-days exposure to 3.5 percent NaCl.

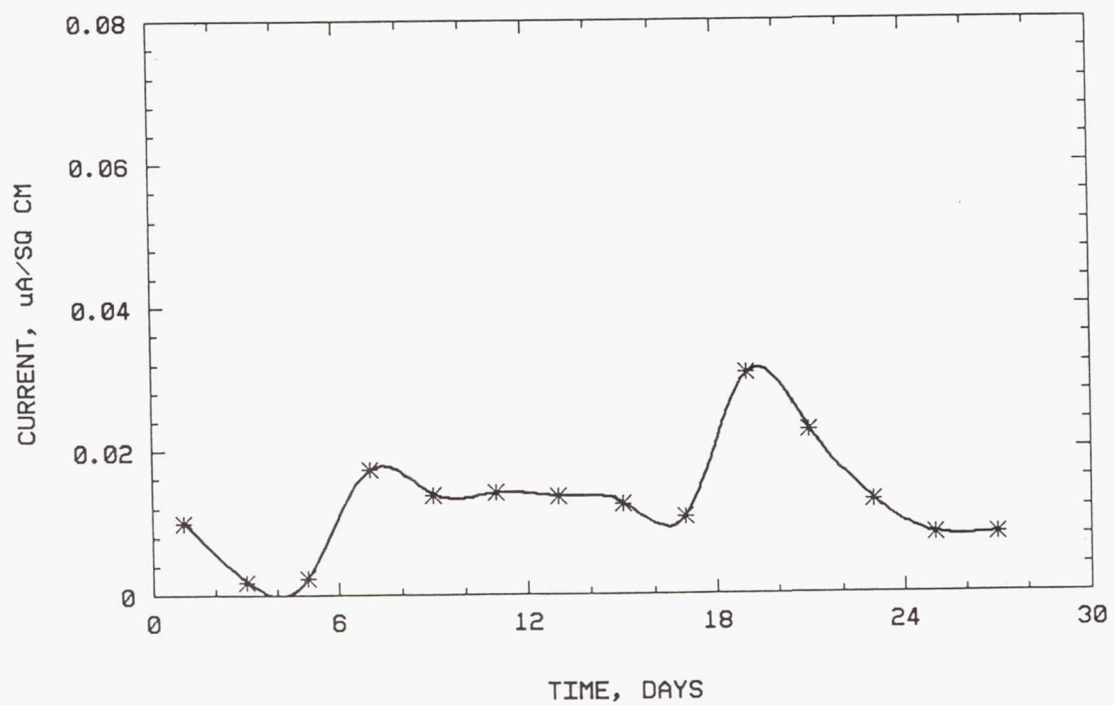


Figure 18. Corrosion current, 2219-T87 aluminum, 1-mil anodized IVD coat.

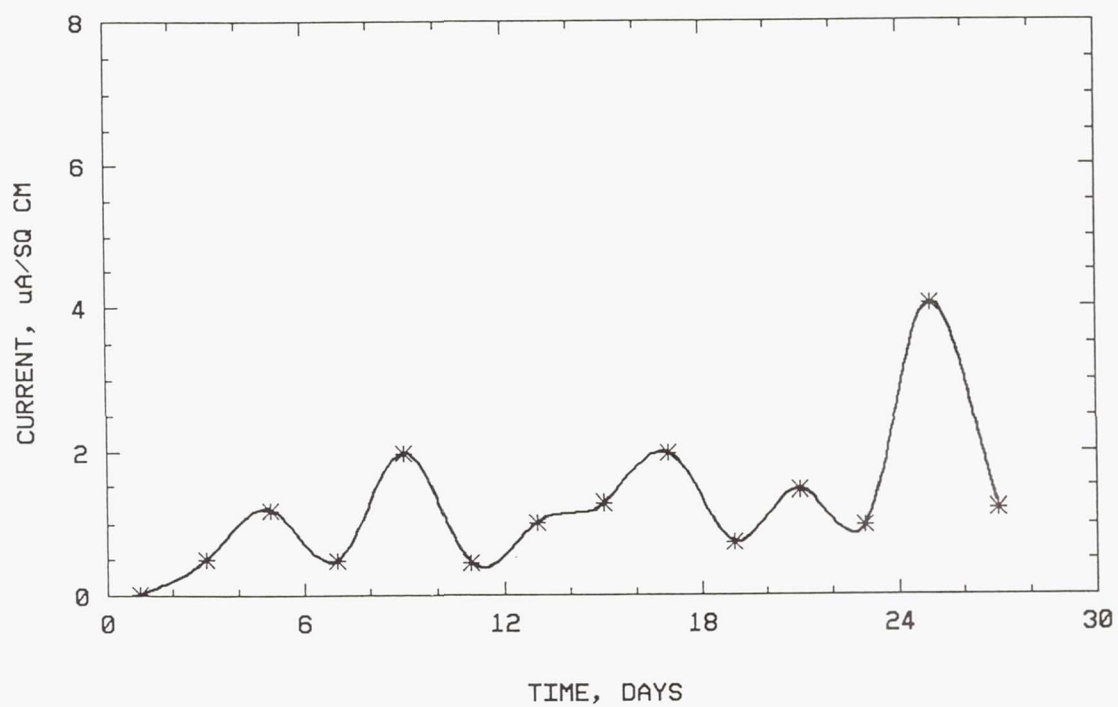


Figure 19. Corrosion current, 2219-T87 aluminum, 1-mil IVD coat.

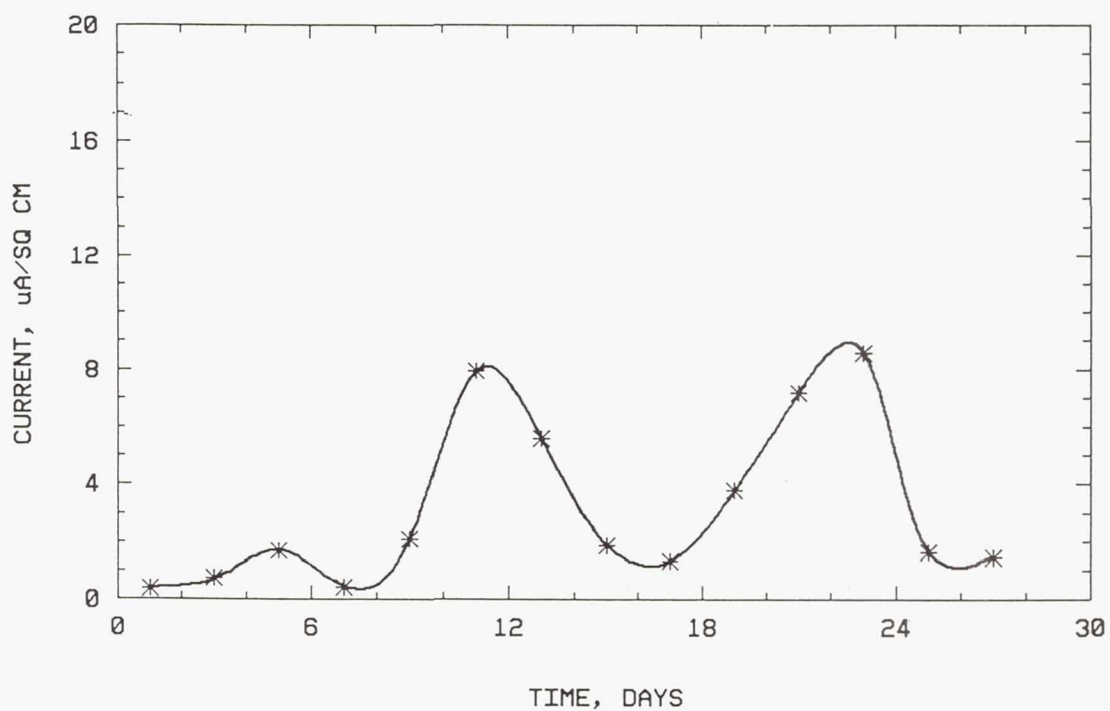


Figure 20. Corrosion current, 7075-T6 aluminum, 1-mil IVD coat.

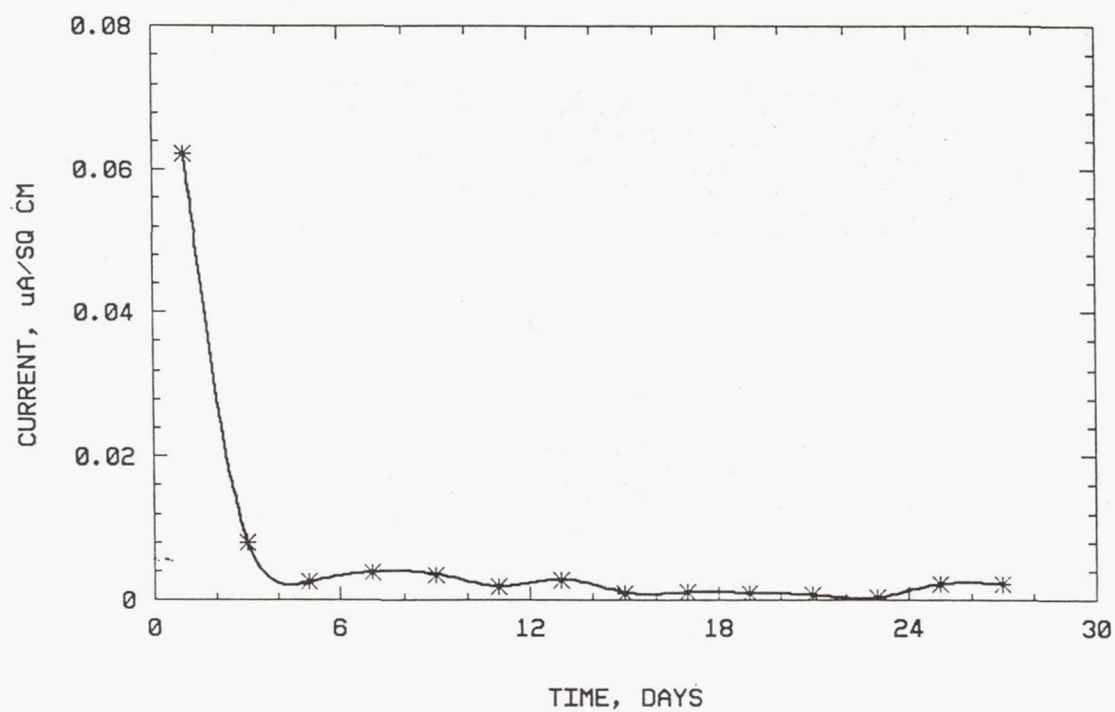


Figure 21. Corrosion current, 7075-T6 aluminum, 2-mil IVD coat anodized.

APPROVAL

THE CORROSION PROTECTION OF METALS BY
ION VAPOR DEPOSITED ALUMINUM

By Merlin D. Danford

The information in this report has been reviewed for technical content. Review of any information concerning the Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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